



STACK SAMPLING REPORT  
FOR  
MERCURY TESTING  
ON THE  
HOLCIM (TEXAS) LP  
MIDLOTHIAN PLANT – MIDLOTHIAN, TEXAS  
KILN NO. 1  
DESULPHURIZATION SCRUBBER INLET DUCT AND STACK

PROJECT NO. 07-043A

OCTOBER 2007

PREPARED FOR:  
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(Total Number of Pages = 715)



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## **EXECUTIVE SUMMARY**

Air Sampling Associates, Inc. of Lewisville, Texas conducted stack testing at the Holcim (Texas) LP, Midlothian Plant, located in Midlothian, Texas during the week of October 8, 2007. The purpose of the stack testing was to determine the amount of mercury being emitted to the atmosphere via the Desulphurization Scrubber Stack and to determine the removal efficiency of mercury by the Desulphurization Scrubber on Kiln No. 1. Set-up and safety training were conducted on October 8, 2007; field recovery and stratification testing were conducted on October 10, 2007; Raw Mill On testing was conducted on October 11, 2007; and Raw Mill Off testing was conducted on October 12, 2007.

The sampling team consisted of Mr. Bill Mullins, Mr. Bill Hefley, Mr. Patrick Selakovich, and Mr. John Stanley. Mr. Mullins was the test team leader.

Dr. Laura Kinner with Emission Monitoring, Inc. was the Project Manager providing coordination of the testing with plant personnel. Mr. Philip Dufresne with Ohio Lumex Company, Inc. provided on-site analyses of the EPA Draft Method 30B samples.

The sampling followed the procedures set forth in Title 40 of the Code of Federal Regulations, Part 60 (40CFR60), Appendix A, Test Methods 1, 2, 3A, 4, and 6C; EPA Draft Method 30B; and Flue Gas Adsorbent Mercury Speciation (FAMS) using multimedia adsorbent tubes. Results are presented in Tables 1 through 12 on pages 5 through 12.

Nine runs total were collected at two operating conditions for the kiln. Four runs were collected during the first operating condition, which was with the Raw Mill On. Five runs were collected during the second operating condition, which was with the Raw Mill Off.



Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test.

Prior to testing, a stratification test was performed at the Scrubber Stack. Sulfur dioxide was used as a surrogate for mercury to verify the absence of stratification across the stack. Due to the low concentrations of sulfur dioxide from the Scrubber Stack and the temporal variation associated with the process, carbon dioxide was also used. A twelve point traverse was conducted with the reference method analyzer sampling system. The sulfur dioxide and carbon dioxide content from each traverse point was normalized for temporal variation and compared to a stationary probe, the kiln CEMS. The results indicated that the stack was not stratified. The stratification results are presented in Appendix I of this report.

Per EPA Draft Method 30B, three field recovery tests were conducted at each sampling location prior to testing to verify the calibration range of the Ohio Lumex Company, Inc. instrumentation, the sampling rate, and the sampling duration of the EPA Draft 30B sampling equipment. An Analytical Bias Test, a Multipoint Analyzer Calibration, and analysis of independent calibration standards were also performed per the method. Appendix G presents the field recovery results and calibration QA/QC.

Four sets of paired EPA Draft Method 30B Sorbent Tubes were sampled simultaneously at the Scrubber Stack and at the Scrubber Inlet Duct with the Raw Mill On and five sets of paired EPA Draft Method 30B Sorbent Tubes were sampled simultaneously at the Scrubber Stack and at the Scrubber Inlet Duct with the Raw Mill Off. Concurrent with the EPA Draft Method 30B samples during each condition, paired FAMS traps were also sampled simultaneously to provide speciated mercury data.

The Sorbent traps for EPA Draft Method 30B were analyzed on site immediately after testing and the FAMS traps were sent to Frontier GeoSciences, Inc. for analyses per EPA Method 1631 Revision E.





Four tests for stack flow rate, oxygen, and carbon dioxide were conducted at the Scrubber Stack and four tests for oxygen were conducted at the Scrubber Inlet Duct with the Raw Mill On. Five tests for stack flow rate, oxygen, and carbon dioxide were conducted at the Scrubber Stack and five tests for oxygen were conducted at the Scrubber Inlet Duct with the Raw Mill Off. The tests were conducted simultaneously with the EPA Draft Method 30B tests and FAMS tests during each condition. Run Nos. 1-4 were sixty minutes in duration and Run Nos. 5-9 were forty-five minutes in duration. The runs during the Raw Mill Off operating condition were shortened due to the kiln not being able to operate at the desired condition for the length of time required to collect sixty minute samples. A shorter run duration did not affect the sample catch due to the elevated concentration levels of mercury at this process condition. Prior to testing, a calibration error test and sampling system bias test were performed on the reference method analyzers at each location.

The in-line raw mill operates roughly 90% of the kiln operating hours. Stack concentrations of mercury ranged from 2.0  $\mu\text{g}/\text{DSCM}$  @ 7%  $\text{O}_2$  to 4.6  $\mu\text{g}/\text{DSCM}$  @ 7%  $\text{O}_2$  for mill on operation which corresponds to a mass emission rate of between 0.6 grams/hr and 1.4 grams/hr at the stack volumetric flow rates measured.

The test results for Raw Mill On operation demonstrate that; 1) the scrubber inlet concentrations are virtually the same as the stack, and, 2) the scrubber converts oxidized mercury to elemental mercury which is re-emitted as seen by the higher concentrations of elemental Hg at the stack versus inlet location. It is likely also that there is hysteresis in the scrubber. When the raw mill is off, large amounts of  $\text{Hg}^{+2}$  are collected in the scrubber which converts back to elemental Hg and re-emits during mill on conditions.

The in-line raw mill is off roughly 10% of the kiln operating hours. The raw mill is off during weekly scheduled preventative maintenance and malfunction. Stack concentrations of mercury compounds ranged from 18.3  $\mu\text{g}/\text{DSCM}$  @ 7%  $\text{O}_2$  to 25.3

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$\mu\text{g/DSCM @ 7\% O}_2$  for mill off operation, which corresponds to a mass emission rate of between 9.1 grams/hr to 12.6 grams/hr at the stack volumetric flow rates measured. Similar mass emissions as raw mill on.

The test results for Raw Mill Off operation demonstrate that; 1) the scrubber inlet concentrations were nearly 40 times higher than with mill on operation, 2) the inlet mercury speciation varies from 1:1 elemental to oxidized in nature to a factor of 3:1, 3) >95% of the mercury compounds are removed by the scrubber, and 4) the in-line raw mill is a passive control device for mercury when operating based on comparisons to mill on data.

Speciated data from FAMS adsorbent tubes during mill off indicated some removal of elemental Hg across the scrubber. Since elemental Hg is not water soluble this was a result which has yet to be explained; however, the data was observed to be very consistent from run to run. FAMS results for total Hg do compare relatively well with total Hg results from the draft Method 30B analyses, so further investigation into this unexpected result is needed.



## SUMMARY OF RESULTS

**Table 1: Summary of Raw Mill On Tests (EPA Methods 1, 2, 3A, and 4)  
– Kiln No. 1 Scrubber Stack**

Run No.	1	2	3	4	Average
Test Date	10/11/07	10/11/07	10/11/07	10/11/07	----
Test Time	1030-1130	1215-1315	1345-1445	1515-1615	----
Flow Rate - DSCFM	364,439	369,560	369,886	350,648	363,633
Stack Temp. - °F	139	139	139	141	140
O <sub>2</sub> - % Vol. Dry	14.0	14.0	14.1	14.1	14.1
CO <sub>2</sub> - % Vol. Dry	11.9	11.7	11.3	11.5	11.6
Moisute Content - %	17.32	17.16	16.91	16.91	17.08
Raw Mill	On	On	On	On	On

**Table 2: Summary of Raw Mill On Tests (EPA Method 3A)  
– Kiln No. 1 Scrubber Inlet Duct**

Run No.	1	2	3	4	Average
Test Date	10/11/07	10/11/07	10/11/07	10/11/07	-----
Test Time	1030-1130	1215-1315	1345-1445	1515-1615	-----
O <sub>2</sub> - % Vol. Dry	13.4	13.5	13.8	13.7	13.6
Raw Mill	On	On	On	On	On



**Table 3: Summary of Raw Mill On Tests (EPA Draft Method 30B)  
– Kiln No. 1 Scrubber Stack**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Total Mercury		
						Total ng	µg/m <sup>3</sup>	µg/m <sup>3</sup> @ 7% O <sub>2</sub>
Mill On	Train 1	10/11/07	1030-	0.027	14.0	60.7	2.3	4.6
Run No. 1	Train 2		1130	0.030		64.4	2.1	4.3
Mill On	Train 1	10/11/07	1215-	0.026	14.0	52.2	2.0	4.0
Run No.2	Train 2		1315	0.031		55.0	1.8	3.6
Mill On	Train 1	10/11/07	1345-	0.027	14.1	32.0	1.2	2.4
Run No. 3	Train 2		1445	0.030		39.1	1.3	2.6
Mill On	Train 1	10/11/07	1515-	0.027	14.1	26.4	1.0	2.0
Run No. 4	Train 2		1615	0.030		34.8	1.1	2.3

**Table 4: Summary of Raw Mill On Tests (EPA Draft Method 30B)  
– Kiln No. 1 Scrubber Inlet Duct**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Total Mercury		
						Total ng	µg/m <sup>3</sup>	µg/m <sup>3</sup> @ 7% O <sub>2</sub>
Mill On	Train 1	10/11/07	1030-	0.028	13.4	86.3	3.1	5.7
Run No. 1	Train 2		1130	0.030		97.4	3.3	6.1
Mill On	Train 1	10/11/07	1215-	0.028	13.5	85.8	3.1	5.8
Run No.2	Train 2		1315	0.030		90.9	3.1	5.8
Mill On	Train 1	10/11/07	1345-	0.027	13.8	51.4	1.9	3.7
Run No. 3	Train 2		1445	0.030		57.3	1.9	3.8
Mill On	Train 1	10/11/07	1515-	0.027	13.7	46.3	1.7	3.4
Run No. 4	Train 2		1615	0.029		49.5	1.7	3.3



**Table 5: Summary of Raw Mill On Tests (FAMS Method) – Kiln No.1 Scrubber Stack**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Particulate Mercury		Elemental Mercury		Oxidized Mercury		Total Mercury	
						ng	µg/m <sup>3</sup>	ng	µg/m <sup>3</sup>	ng	µg/m <sup>3</sup>	Total ng	µg/m <sup>3</sup> @ 7% O <sub>2</sub>
Mill On Run No. 1	Train 1 Train 2	10/11/07	1030-1130	0.016 0.019	14.0	0.2 0.3	0.01 0.01	34.6 39.6	2.22 2.04	0.1 0.1	0.00 0.00	34.8 40.0	2.24 2.05
Mill On Run No.2	Train 1 Train 2	10/11/07	1215-1315	0.019 0.019	14.0	0.1 0.2	0.00 0.01	33.1 31.8	1.78 1.67	0.0 0.0	0.00 0.00	33.2 32.0	1.78 1.68
Mill On Run No. 3	Train 1 Train 2	10/11/07	1345-1445	0.018 0.019	14.1	0.1 2.8	0.00 0.15	20.6 20.8	1.13 1.10	0.0 0.3	0.00 0.01	20.7 23.9	1.14 1.26
Mill On Run No. 4	Train 1 Train 2	10/11/07	1515-1615	0.018 0.019	14.1	0.0 0.1	0.00 0.01	19.9 20.8	1.09 1.09	0.5 0.3	0.03 0.02	20.4 21.3	1.12 1.11

**Table 6: Summary of Raw Mill On Tests (FAMS Method) – Kiln No. 1 Scrubber Inlet Duct**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Particulate Mercury		Elemental Mercury		Oxidized Mercury		Total Mercury	
						ng	µg/m <sup>3</sup>	ng	µg/m <sup>3</sup>	ng	µg/m <sup>3</sup>	Total ng	µg/m <sup>3</sup> @ 7% O <sub>2</sub>
Mill On Run No. 1	Train 1 Train 2	10/11/07	1030-1130	0.017 0.015	13.4	0.2 0.1	0.01 0.01	41.3 38.1	2.42 2.47	6.9 8.4	0.40 0.54	48.3 46.6	2.83 3.01
Mill On Run No.2	Train 1 Train 2	10/11/07	1215-1315	0.017 0.015	13.5	0.5 0.3	0.03 0.02	30.4 27.5	1.83 1.83	11.8 14.2	0.71 0.94	42.6 41.9	2.57 2.79
Mill On Run No. 3	Train 1 Train 2	10/11/07	1345-1445	0.016 0.015	13.8	0.3 0.3	0.02 0.02	15.1 16.7	0.95 1.13	14.0 11.6	0.88 0.78	29.4 28.6	1.85 1.93
Mill On Run No. 4	Train 1 Train 2	10/11/07	1515-1615	0.017 0.015	13.7	0.4 0.7	0.02 0.05	15.2 14.2	0.92 0.92	10.0 10.1	0.60 0.65	25.6 24.9	1.54 1.61





**Table 7: Summary of Raw Mill Off Tests (EPA Methods 1, 2, 3A, and 4)  
– Kiln No.1 Scrubber Stack**

Run No.	5	6	7	8	9	Average
Test Date	10/12/07	10/12/07	10/12/07	10/12/07	10/12/07	-----
Test Time	0800-0845	0915-1000	1036-1121	1147-1232	1300-1345	-----
Flow Rate - DSCFM	301,087	283,235	288,492	280,925	308,740	292,496
Stack Temp. - °F	142	145	146	145	142	144
O <sub>2</sub> - % Vol. Dry	11.7	11.6	11.8	12.2	13.0	12.1
CO <sub>2</sub> - % Vol. Dry	15.2	15.3	15.3	14.6	13.4	14.8
Moisture Content - %	19.87	20.85	20.69	19.29	18.38	19.82
Raw Mill	Off	Off	Off	Off	Off	Off

**Table 8: Summary of Raw Mill Off Tests (EPA Method 3A)  
– Kiln No. 1 Scrubber Inlet Duct**

Run No.	5	6	7	8	9	Average
Test Date	10/12/07	10/12/07	10/12/07	10/12/07	10/12/07	----
Test Time	0800-0845	0915-1000	1036-1121	1147-1232	1300-1345	----
O <sub>2</sub> - % Vol. Dry	10.8	10.7	10.9	11.4	12.4	11.2
Raw Mill	Off	Off	Off	Off	Off	Off



**Table 9: Summary of Raw Mill Off Tests (EPA Draft Method 30B) –  
Kiln No. 1 Scrubber Stack**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Total Mercury		
						Total ng	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$ @ 7% O <sub>2</sub>
Mill Off	Train 1	10/12/07	0800-	0.025	11.7	298.1	12.1	18.3
Run No. 5	Train 2		0845	0.027		352.8	13.3	20.1
Mill Off	Train 1	10/12/07	0915-	0.025	11.6	376.8	15.3	22.9
Run No. 6	Train 2		1000	0.027		386.2	14.4	21.5
Mill Off	Train 1	10/12/07	1036-	0.024	11.8	400.6	16.5	25.3
Run No. 7	Train 2		1121	0.027		405.1	14.8	22.6
Mill Off	Train 1	10/12/07	1147-	0.024	12.2	376.9	15.7	25.0
Run No. 8	Train 2		1232	0.027		373.2	13.6	21.7
Mill Off	Train 1	10/12/07	1300-	0.025	13.0	322.3	13.1	23.1
Run No. 9	Train 2		1345	0.028		357.6	12.8	22.6

**Table 10: Summary of Raw Mill Off Tests (EPA Draft Method 30B) –  
Kiln No. 1 Scrubber Inlet Duct**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Total Mercury		
						Total ng	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$ @ 7% O <sub>2</sub>
Mill Off	Train 1	10/12/07	0800-	0.025	10.8	3557.1	141.7	195.0
Run No. 5	Train 2		0845	0.027		3748.0	138.9	191.2
Mill Off	Train 1	10/12/07	0915-	0.025	10.7	3348.3	134.8	183.6
Run No. 6	Train 2		1000	0.027		3631.0	135.9	185.2
Mill Off	Train 1	10/12/07	1036-	0.024	10.9	3473.0	141.8	197.1
Run No. 7	Train 2		1121	0.027		3497.0	130.5	181.4
Mill Off	Train 1	10/12/07	1147-	0.024	11.4	3270.0	136.0	199.0
Run No. 8	Train 2		1232	0.026		3122.0	118.6	173.5
Mill Off	Train 1	10/12/07	1300-	0.023	12.4	2230.0	95.5	156.2
Run No. 9	Train 2		1345	0.026		2404.8	92.0	150.4



**Table 11: Summary of Raw Mill Off Tests (FAMS Method) – Kiln No. 1 Scrubber Stack**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Particulate Mercury			Elemental Mercury			Oxidized Mercury			Total Mercury		
						ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	Total ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>
Mill Off Run No. 5	Train 1	10/12/07	0800-0845	0.022	11.7	0.3	0.01	0.02	218.6	10.16	15.35	0.0	0.00	0.00	218.9	10.18	15.4
	Train 2			0.022		0.4	0.02	0.03	240.3	11.17	16.88	0.0	0.00	0.00	240.6	11.19	16.9
Mill Off Run No. 6	Train 1	10/12/07	0915-1000	0.018	11.6	0.3	0.02	0.03	230.8	12.70	18.98	0.0	0.00	0.00	231.1	12.71	19.0
	Train 2			0.022		0.5	0.02	0.03	268.9	12.00	17.94	0.6	0.03	0.04	270.1	12.05	18.0
Mill Off Run No. 7	Train 1	10/12/07	1036-1121	0.022	11.8	0.2	0.01	0.01	243.4	11.19	17.09	0.4	0.02	0.02	243.9	11.21	17.1
	Train 2			0.020		0.2	0.01	0.01	251.0	12.48	19.07	0.1	0.00	0.01	251.3	12.49	19.1
Mill Off Run No. 8	Train 1	10/12/07	1147-1232	0.023	12.2	0.2	0.01	0.01	228.2	10.08	16.10	0.1	0.00	0.00	228.4	10.09	16.1
	Train 2			0.023		0.3	0.01	0.02	289.9	12.55	20.05	0.3	0.01	0.02	290.4	12.57	20.1
Mill Off Run No. 9	Train 1	10/12/07	1300-1345	0.022	13.0	0.2	0.01	0.01	226.6	10.31	18.14	0.2	0.01	0.02	227.0	10.33	18.2
	Train 2			0.021		0.1	0.01	0.01	229.9	11.12	18.40	0.0	0.00	0.00	223.0	11.13	19.6

**Table 12: Summary of Raw Mill Off Tests (FAMS Method) – Kiln No. 1 Scrubber Inlet Duct**

Run No.	Sample ID	Date	Time	Sample Vol. (DSCM)	Oxygen (% Vol.)	Particulate Mercury			Elemental Mercury			Oxidized Mercury			Total Mercury		
						ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>	Total ng	µg/m <sup>3</sup>	@ 7% O <sub>2</sub>
Mill Off Run No. 5	Train 1	10/12/07	0800-0845	0.022	10.8	10.0	0.46	0.64	1043.4	48.24	66.39	1421.6	65.72	90.45	2475.0	114.4	157.5
	Train 2			0.020		7.9	0.39	0.54	1102.5	54.33	74.89	1081.1	53.28	73.44	2191.5	108.2	148.9
Mill Off Run No. 6	Train 1	10/12/07	0915-1000	0.021	10.7	7.9	0.38	0.51	1481.5	70.43	95.96	890.0	42.31	57.65	2379.4	113.1	154.1
	Train 2			0.020		3.2	0.16	0.21	1395.0	68.36	93.14	870.5	42.65	58.12	2268.7	111.2	151.5
Mill Off Run No. 7	Train 1	10/12/07	1036-1121	0.021	10.9	0.0	0.00	0.00	1628.0	77.91	108.28	837.0	40.05	55.67	2465.0	117.9	163.9
	Train 2			0.020		3.1	0.16	0.22	1269.4	62.65	87.09	1044.9	51.57	71.69	2317.4	114.4	159.0
Mill Off Run No. 8	Train 1	10/12/07	1147-1232	0.021	11.4	3.8	0.18	0.27	1592.8	75.93	111.08	691.9	32.98	48.25	2288.5	109.1	159.6
	Train 2			0.020		2.3	0.12	0.17	1600.5	80.95	118.45	605.4	30.62	44.80	2208.3	111.7	163.4
Mill Off Run No. 9	Train 1	10/12/07	1300-1345	0.020	12.4	1.5	0.07	0.12	1019.2	50.72	82.92	599.8	29.85	48.80	1620.4	80.6	131.8
	Train 2			0.020		2.5	0.13	0.21	1127.9	57.82	94.54	516.5	26.47	43.29	1646.8	84.4	138.0



## **DISCUSSION OF SAMPLING RESULTS**

### **Kiln No. 1 Scrubber Stack – Raw Mill On**

#### **Stratification Test**

Prior to testing, a stratification test was performed at the Scrubber Stack. Sulfur dioxide was used as a surrogate for mercury to verify the absence of stratification across the stack. Due to the low concentrations of sulfur dioxide from the Scrubber Stack and the temporal variation associated with the process, carbon dioxide was also used. A twelve point traverse was conducted with the reference method analyzer sampling system. The sulfur dioxide and carbon dioxide content from each traverse point was normalized for temporal variation and compared to a stationary probe, the kiln CEMS. The results indicated that the stack was not stratified. The stratification results are presented in Appendix I of this report.

The stratification tests for sulfur dioxide appeared to be valid representations of the actual emissions during the tests. All leak checks performed on the reference method analyzer sampling system showed no leaks before or after testing. The calibration error test on the sulfur dioxide analyzer was valid with no variations greater than 0.66% compared to the allowed 2.0% calibration error. The calibration drift test performed at the completion of the test was stable with no variations greater than 2.60% compared to the allowed 3.0% calibration drift. The bias test was valid with no bias results greater than 2.50% compared to the allowed 5.0% system bias.

The stratification tests for carbon dioxide appeared to be valid representations of the actual emissions during the tests. All leak checks performed on the reference method analyzer sampling system showed no leaks before or after testing. The calibration error test on the carbon dioxide analyzer was valid with no variations greater than 1.66% compared to the allowed 2.0% calibration error. The calibration drift test performed at 07-043A



the completion of the test was stable with no variations greater than 0.55% compared to the allowed 3.0% calibration drift. The bias test was valid with no bias results greater than 0.50% compared to the allowed 5.0% system bias.

#### **Flow Rate**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for flow rate appeared to be a valid representation of the actual stack flow rate during the tests. All leak checks performed on the reference method sampling train and pitot tubes showed no leaks before or after testing. The indicative parameters of the tests were in close agreement. The measured moisture contents (%M) were within 1.43% of the mean value. The measured flow rates (DSCFM) were within 3.57% of the mean value.

#### **Oxygen**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for O<sub>2</sub> appeared to be a valid representation of the actual emissions during the tests. The calibration error test on the O<sub>2</sub> analyzer was valid with no variations greater than 0.40%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.64%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 1.08%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of O<sub>2</sub> for the four tests showed a range of -0.36 percent to +0.36 percent variation from the mean value of 14.1% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).

#### **Carbon Dioxide**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for CO<sub>2</sub> appeared to be a valid





representation of the actual emissions during the tests. The calibration error test on the CO<sub>2</sub> analyzer was valid with no variations greater than 1.66%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.50%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 0.60%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of CO<sub>2</sub> for the four tests showed a range of -2.59 percent to +2.59 percent variation from the mean value of 11.6% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).

#### **Total Mercury (EPA Draft Method 30B)**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for mercury using EPA Draft Method 30B appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. All field recovery results were well within the EPA Draft Method 30B criteria. Agreement between run pairs was also within method criteria without any signs of mercury breakthrough from section A to B of the traps.

Statistical analysis of the sampling results will not be discussed in this report.

#### **Speciated Mercury (FAMS Method)**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for mercury using the FAMS Method appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. The results demonstrated good agreement between the paired samples. Speciated mercury results demonstrate that the primary species is elemental in nature.



Run 6 pairs demonstrated an 88% elemental to total mercury ratio and runs 1-5 demonstrated elemental to total mercury of 88-93%.

Statistical analysis of the sampling results will not be discussed in this report.

### **Kiln No. 1 Scrubber Inlet Duct – Raw Mill On**

#### **Oxygen**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for O<sub>2</sub> appeared to be a valid representation of the actual emissions during the tests. The calibration error test on the O<sub>2</sub> analyzer was valid with no variations greater than 0.36%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.12%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 0.48%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of O<sub>2</sub> for the four tests showed a range of -1.47 percent to +1.47 percent variation from the mean value of 13.6% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).

#### **Total Mercury (EPA Draft Method 30B)**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for mercury using EPA Draft Method 30B appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. All field recovery results were well within the EPA Draft Method 30B criteria. Agreement between run pairs was also within method criteria without any signs of mercury breakthrough from section A to B of the traps.





Results showed about 20-40% removal of total mercury across the scrubber. These results have no error analyses applied.

Statistical analysis of the sampling results will not be discussed in this report.

#### **Speciated Mercury (FAMS Method)**

Only four tests were taken at the Raw Mill On operating condition because the Raw Mill failed to operate for the fifth test. The four tests for mercury using the FAMS Method appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. The results demonstrated good agreement between the paired samples. Speciated mercury results demonstrate that the primary species during runs 2 and 3 is elemental in nature, and that there is more elemental mercury at the stack than at the inlet location which corresponds to mercury re-emission from conversion of collected oxidized mercury.

Statistical analysis of the sampling results will not be discussed in this report.

### **Kiln No. 1 Scrubber Stack – Raw Mill Off**

#### **Flow Rate**

The five tests for flow rate appeared to be a valid representation of the actual stack flow rate during the tests. All leak checks performed on the reference method sampling train and pitot tubes showed no leaks before or after testing. The indicative parameters of the tests were in close agreement. The measured moisture contents (%M) were within 7.25% of the mean value. The measured flow rates (DSCFM) were within 5.55% of the mean value.

### Oxygen

The five tests for O<sub>2</sub> appeared to be a valid representation of the actual emissions during the tests. The calibration error test on the O<sub>2</sub> analyzer was valid with no variations greater than 0.24%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.24%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 0.96%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of O<sub>2</sub> for the five tests showed a range of -3.81 percent to +7.99 percent variation from the mean value of 12.1% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).

### Carbon Dioxide

The five tests for CO<sub>2</sub> appeared to be a valid representation of the actual emissions during the tests. The calibration error test on the CO<sub>2</sub> analyzer was valid with no variations greater than 1.75%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.70%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 1.00%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of CO<sub>2</sub> for the five tests showed a range of -9.21 percent to +3.66 percent variation from the mean value of 14.8% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).

### Total Mercury (EPA Draft Method 30B)

The five tests for mercury using EPA Draft Method 30B appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. All field



recovery results were well within the EPA Draft Method 30B criteria. Agreement between run pairs was also within method criteria without any signs of mercury breakthrough from section A to B of the traps.

Statistical analysis of the sampling results will not be discussed in this report.

#### **Speciated Mercury (FAMS Method)**

The five tests for mercury using the FAMS Method appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. The results demonstrated good agreement between the paired samples. Speciated mercury results demonstrate an approximate 50:50 ratio of elemental Hg to oxidized Hg.

Statistical analysis of the sampling results will not be discussed in this report.

### **Kiln No. 1 Scrubber Inlet Duct – Raw Mill Off**

#### **Oxygen**

The five tests for O<sub>2</sub> appeared to be a valid representation of the actual emissions during the tests. The calibration error test on the O<sub>2</sub> analyzer was valid with no variations greater than 0.36%, compared to the allowed 2.0% calibration error. The calibration drift tests performed at the completion of each run were stable with no variations greater than 0.12%, compared to the allowed 3.0% calibration drift. The bias tests were valid with no bias results greater than 0.36%, compared to the allowed 5.0% system bias.

The concentrations (% Vol. dry) of O<sub>2</sub> for the five tests showed a range of -4.80 percent to +10.32 percent variation from the mean value of 11.2% Vol. dry. The concentrations were adjusted with equation 7E-5 (40CFR60, Appendix A, Method 7-E).





#### **Total Mercury (EPA Draft Method 30B)**

The five tests for mercury using EPA Draft Method 30B appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. All field recovery results were well within the EPA Draft Method 30B criteria. Agreement between run pairs was also within method criteria without any signs of mercury breakthrough from section A to B of the traps. Stack concentrations of mercury were observed to be about 6 times higher than with mill on.

Statistical analysis of the sampling results will not be discussed in this report.

#### **Speciated Mercury (FAMS Method)**

The five tests for mercury using the FAMS Method appeared to be a valid representation of the actual emissions during the tests. All leak checks performed on the reference method sampling train showed no leaks before or after testing. The results demonstrated good agreement between the paired samples. Speciated mercury results demonstrate that elemental to oxidized ratios are about 3:1. The inlet concentrations observed during mill off conditions are similar to other kilns tested. The concentration levels were about a factor of 40 times higher than mill on conditions.

Statistical analysis of the sampling results will not be discussed in this report.



## **DESCRIPTION OF SAMPLING LOCATION**

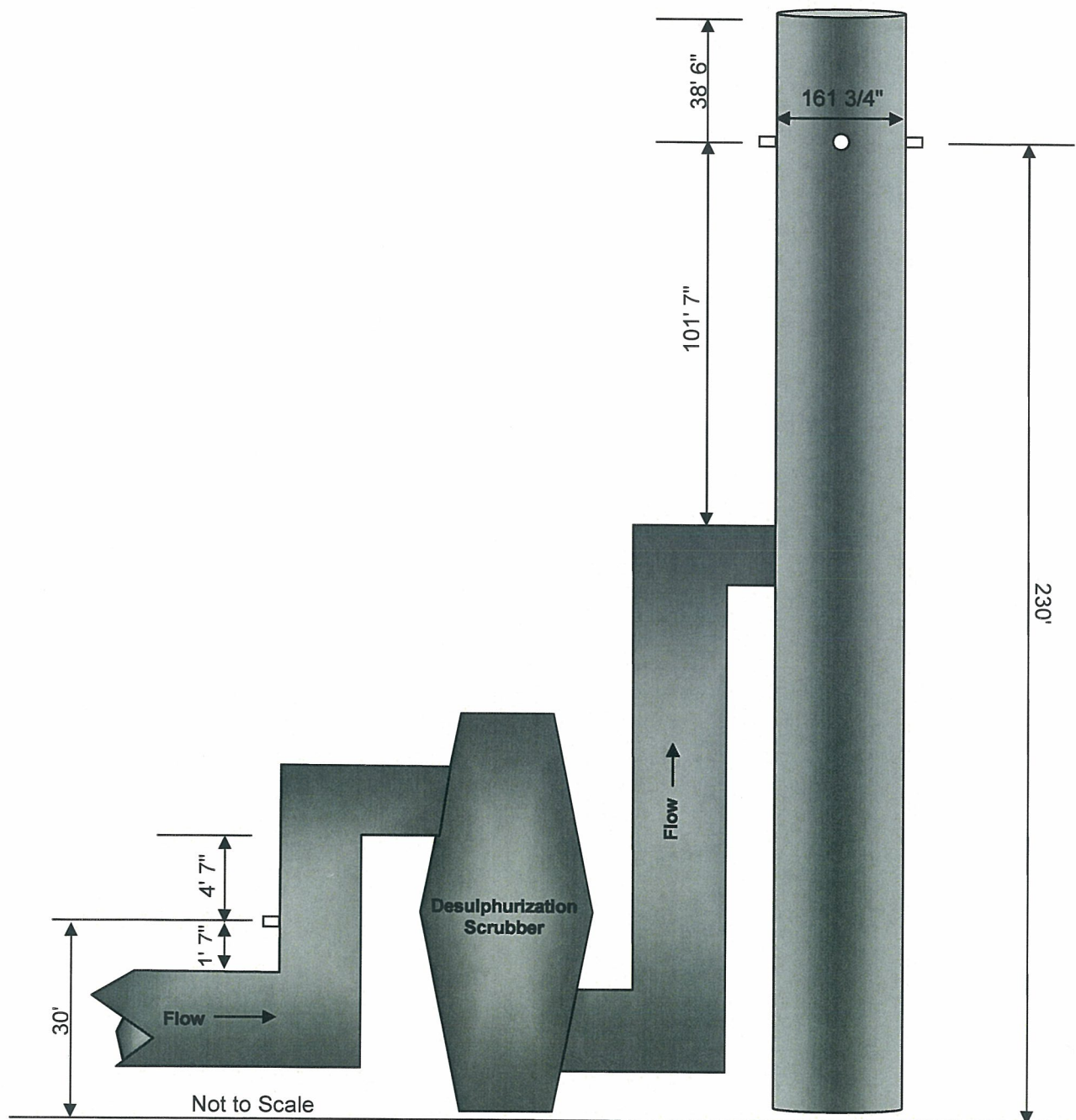
The sampling ports on the Kiln No. 1 Scrubber Stack are located approximately 230 feet above the ground. The sampling ports are located 101 feet 7 inches (7.54 stack diameters) downstream from the inlet to the stack and 38 feet 6 inches (2.86 stack diameters) upstream from the outlet of the stack.

The sampling ports on the Kiln No. 1 Scrubber Inlet Duct are located approximately 30 feet above the ground. The sampling ports are located 1 foot 7 inches (0.13 equivalent duct diameters) downstream from an expansion in the duct and 4 feet 7 inches (0.38 equivalent duct diameters) upstream from a bend in the duct.



## DESCRIPTION OF SAMPLING LOCATION

Figure 1: Kiln No. 1 Scrubber Inlet Duct and Stack





## **SAMPLING AND ANALYTICAL PROCEDURES**

The sampling followed the procedures set forth in 40CFR60, Appendix A, Test Methods 1, 2, 3A, 4, and 6C; EPA Draft Method 30B; and Flue Gas Adsorbent Mercury Speciation (FAMS).

### **Flow Rate**

The stack velocity was determined according to EPA Methods 1 and 2. A preliminary velocity traverse was made at each of two ports on the stack in order to determine the uniformity and magnitude of the flow prior to testing. All traverse points were checked for cyclonic flow and the average angle of cyclonic flow was 1 degree. Six traverse points were sampled from each of two ports for a total of twelve velocity traverse points.

The pitot tube lines were checked for leaks before and after each test under a vacuum and a pressure. The lines were also checked for clearance and the manometer was zeroed before each test.

The stack moisture samples were taken according to EPA Method 4. Samples of sixty minute duration were taken at a single traverse point (Port C, Point No. 3) during Run Nos. 1-4. Samples of forty-five minute duration were taken at a single traverse point (Port B, Point No. 3) during Run Nos. 5-9. Data was recorded at five minute intervals.

The moisture sampling train was leak checked at the end of the sampling probe at fifteen inches of mercury vacuum before each test, and again at the conclusion of each test at the highest vacuum recorded during sampling. This was done to predetermine the possibility of a diluted sample.





The 'front-half' of the moisture sampling train contained the following components:

Heated Glass lined probe @ 248°F ± 25°F

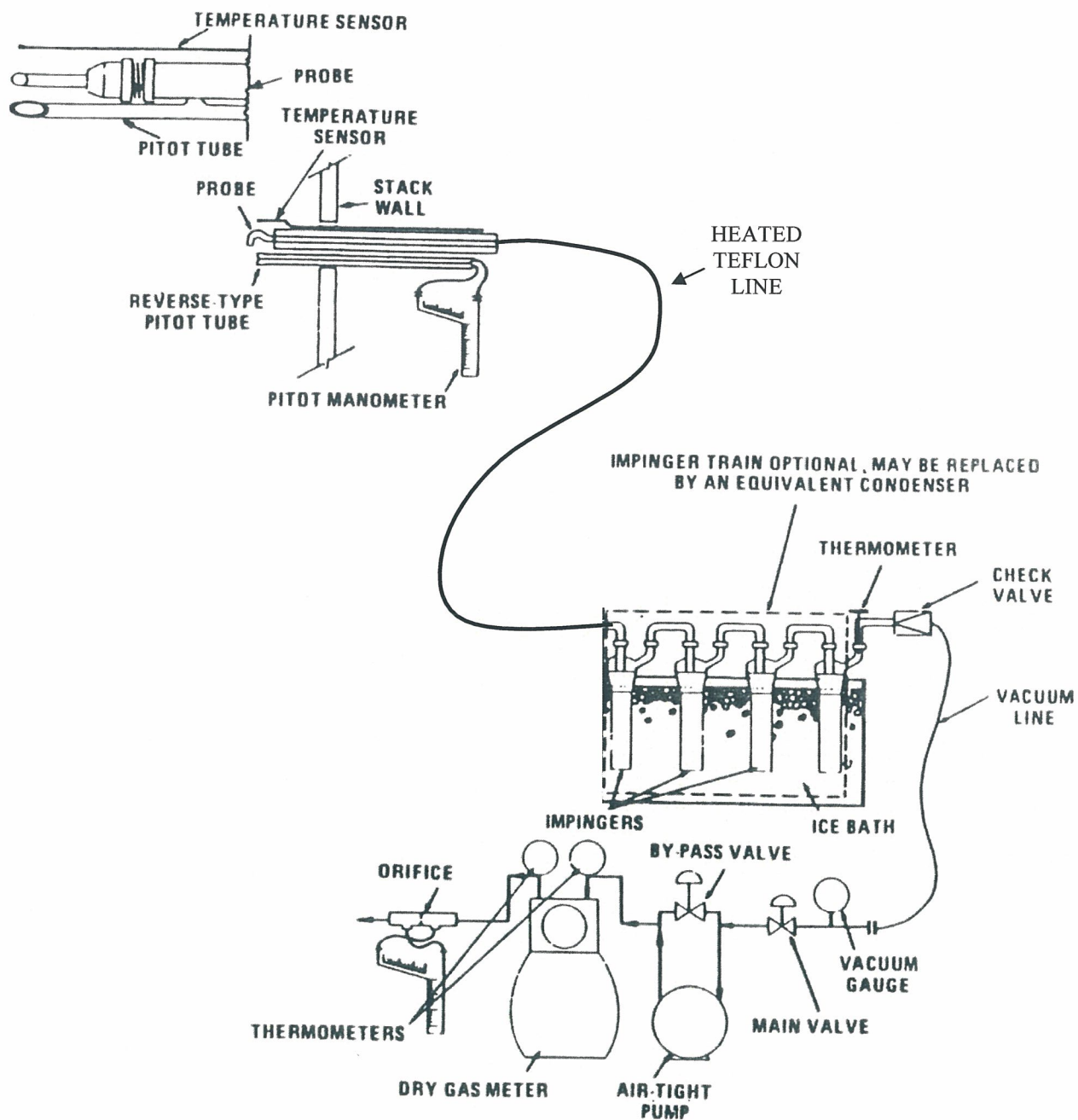
Heated Teflon line @ 248°F ± 25°F

The 'back-half' of the moisture sampling train contained the following components:

**Table 13: Reference Method 4 Sampling Train**

<b>Impinger No.</b>	<b>Impinger Type</b>	<b>Impinger Contents</b>	<b>Amount</b>	<b>Parameter Collected</b>
<b>1</b>	Modified	D.I. Water	100 ml	H <sub>2</sub> O
<b>2</b>	Greenburg-Smith	D.I. Water	100 ml	H <sub>2</sub> O
<b>3</b>	Modified	Empty	-----	H <sub>2</sub> O
<b>4</b>	Modified	Silica Gel	250 g	H <sub>2</sub> O

**Figure 2: EPA Methods 1, 2, 3, and 4 Sampling Train**







### **Sulfur Dioxide**

The sulfur dioxide sampling during the stratification test was performed according to EPA Method 6C. A Western Research Series 900 sulfur dioxide analyzer (serial no. AC-921-9440-1) was used to monitor the concentrations of sulfur dioxide during each run. The reference method analyzer was operated at a range of 0 to 100 ppm. A multi-point calibration was performed on the reference method analyzer prior to testing. After each run, the zero and calibration drift of the reference method analyzer was checked. The calibration gases were as follows:

#### **Zero Gas**

45.9 ppm Sulfur Dioxide in Nitrogen (ALM064315)

90.5 ppm Sulfur Dioxide in Nitrogen (ALM006163)

EPA Protocol Gas Certificates of Analysis for the calibration gases are included in Appendix F.

The reference method sampling system consisted of a heated stainless steel probe, a moisture removal system, and a Teflon sample line. Calibration gases for the bias and drift checks were introduced at the outlet of the heated stainless steel probe. Samples of two minute duration were taken at each of twelve traverse points. The reference method analyzer sampling system was leak-checked at the end of the sampling probe at fifteen inches of mercury vacuum prior to and at the conclusion of testing.

### **Oxygen**

Oxygen sampling was performed according to EPA Method 3A. A Servomex Model 1440D oxygen analyzer (serial no. 1420D/3279) was used to monitor the concentrations of oxygen at the Scrubber Stack during each run. A M&C Model PMA100 oxygen analyzer (serial no. 0502218) was used to monitor the concentrations of oxygen at the Scrubber Inlet Duct during each run. The reference method analyzers were operated at ranges of 0 to 25%. A multi-point calibration was performed on the reference method  
07-043A



analyzers prior to testing. After each run, the zero and calibration drift of the reference method analyzers were checked. The calibration gases were as follows:

Zero Gas

12.20% Oxygen in Nitrogen (ALM021252)

22.30% Oxygen in Nitrogen (ALM002541)

EPA Protocol Gas Certificates of Analysis for the calibration gases are included in Appendix F.

The reference method sampling system at the Scrubber Stack consisted of a heated stainless steel probe, a moisture removal system, and a Teflon sample line. The reference method sampling system at the Scrubber Inlet Duct consisted of a heated stainless steel probe, a heated Teflon line, and a moisture removal system. Calibration gases for the bias and drift checks were introduced at the outlet of each sampling probe. Samples of sixty minute duration were taken at a single traverse point (Port D, Point No. 3 at the stack and Port G Centroid Point at the inlet duct) during Run Nos. 1-4. Samples of forty-five minute duration were taken at a single traverse point (Port D, Point No. 3 at the stack and Port G Centroid Point at the inlet duct) during Run Nos. 5-9. The reference method analyzer sampling systems were leak-checked at the end of the sampling probes at fifteen inches of mercury vacuum prior to and at the conclusion of testing.

**Carbon Dioxide**

Carbon Dioxide sampling was performed according to EPA Method 3A. A Servomex Model 1440D carbon dioxide analyzer (serial no. 1415D/3279) was used to monitor the concentrations of carbon dioxide at the Scrubber Stack during each run. The reference method analyzer was operated at a range of 0 to 20%. A multi-point calibration was performed on the reference method analyzer prior to testing. After each run, the zero





and calibration drift of the reference method analyzer was checked. The calibration gases were as follows:

Zero Gas

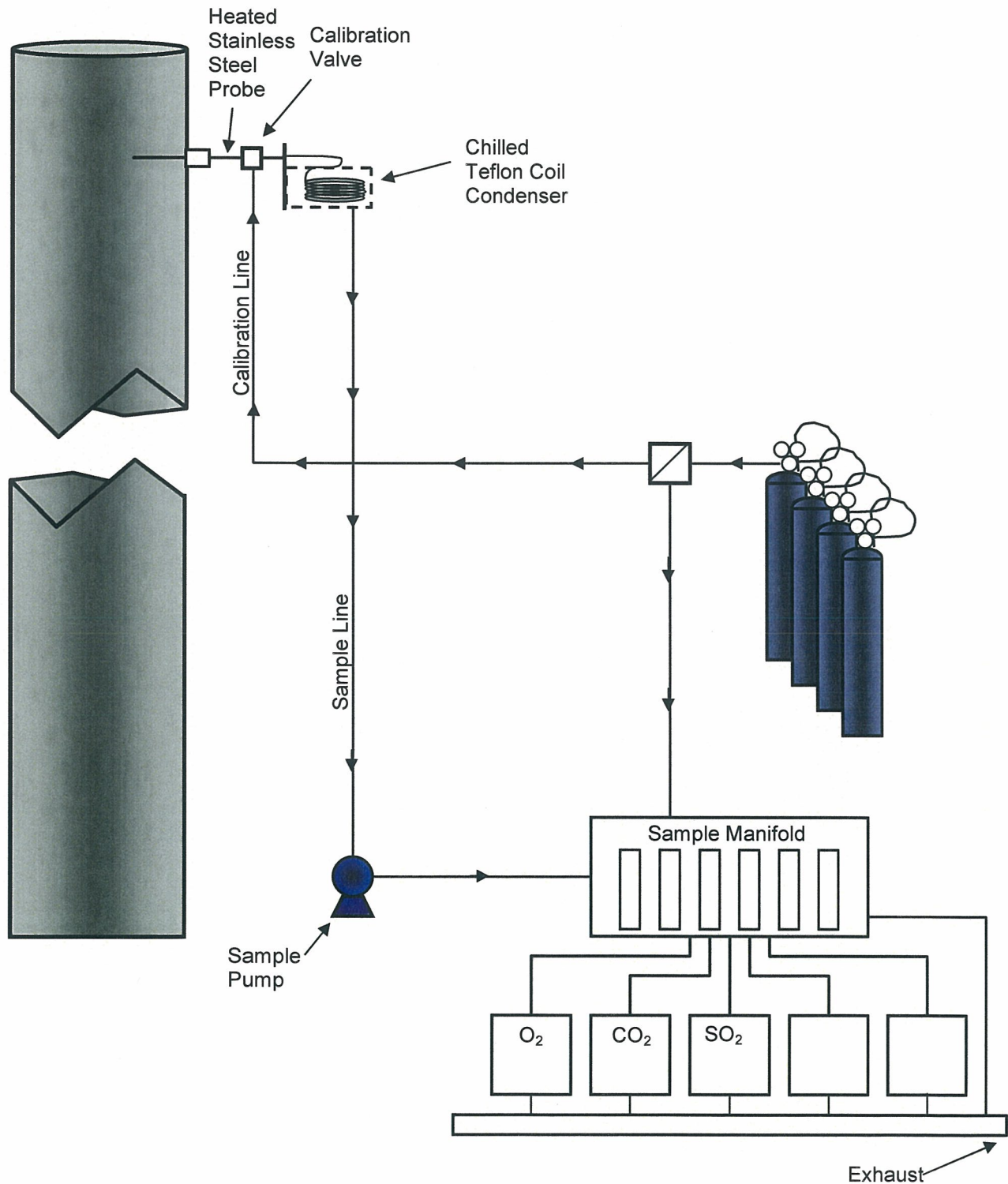
10.10% Carbon Dioxide in Nitrogen (ALM021252)

18.10% Carbon Dioxide in Nitrogen (ALM002541)

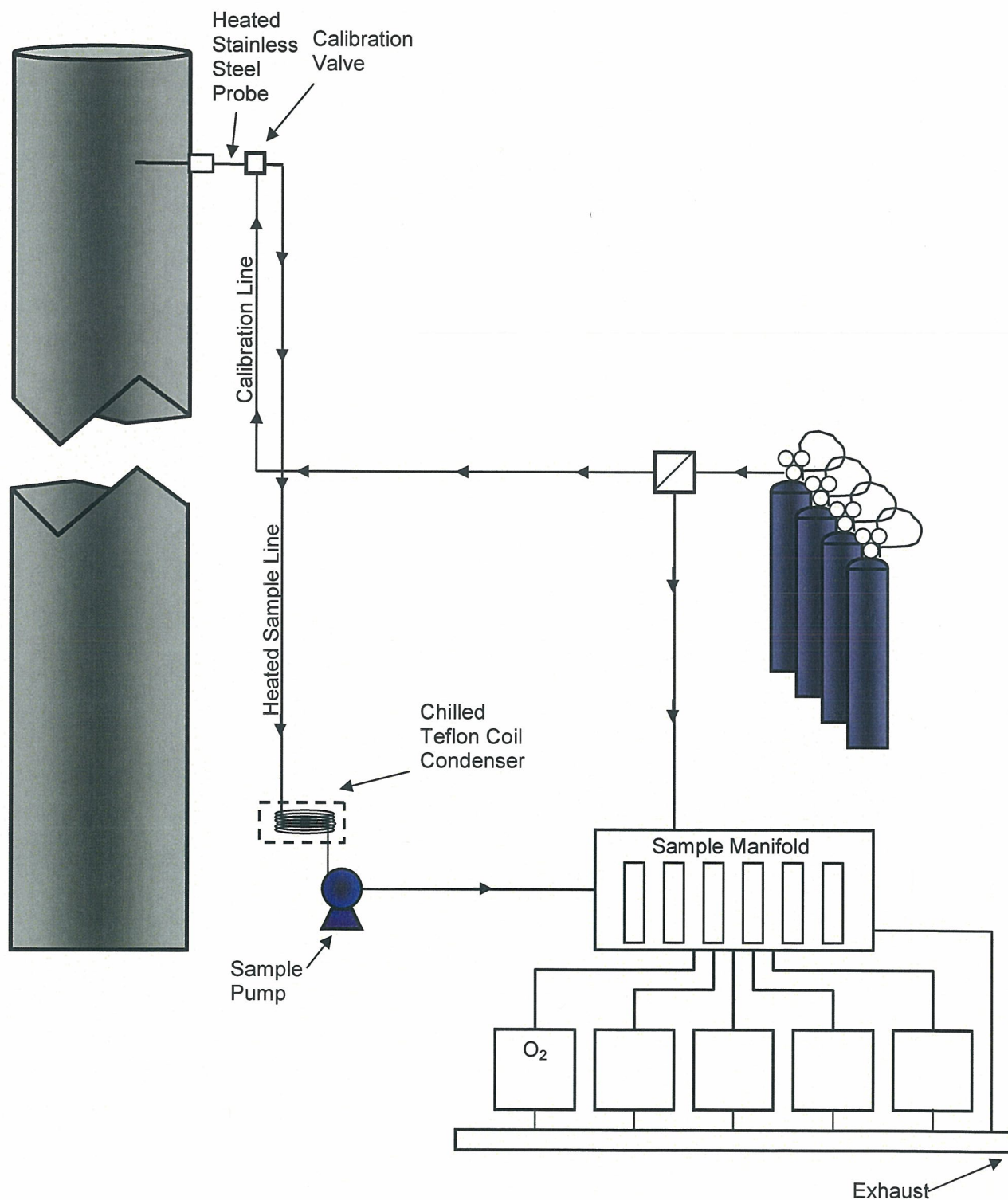
EPA Protocol Gas Certificates of Analysis for the calibration gases are included in Appendix F.

The reference method sampling system at the Scrubber Stack consisted of a heated stainless steel probe, a moisture removal system, and a Teflon sample line. Calibration gases for the bias and drift checks were introduced upstream of the moisture removal system. Samples of sixty minute duration were taken at a single traverse point (Port D, Point No. 3) during Run Nos. 1-4. Samples of forty-five minute duration were taken at a single traverse point (Port D, Point No. 3) during Run Nos. 5-9. The reference method analyzer sampling system was leak-checked at the end of the sampling probe at fifteen inches of mercury vacuum prior to and at the conclusion of testing.

**Figure 3: EPA Method 3A and 6C Sampling System – Kiln No. 1 Scrubber Stack**



**Figure 4: EPA Method 3A Sampling System – Kiln No. 1 Scrubber Inlet Duct**







### Total Mercury

Total mercury sampling was performed according to EPA Draft Method 30B. Prior to sampling, an Analytical Bias Test, a Multipoint Analyzer Calibration, and analysis of independent calibration standards were performed on the Ohio Lumex Company, Inc. analyzer. Calibration checks were periodically analyzed in the field per the method. Three paired field recovery samples were also collected simultaneously at the Scrubber Stack and at the Scrubber Inlet Duct at a sampling rate of approximately 0.500 liters per minute for 60 minutes. Each pair consisted of a spiked Sorbent trap and an un-spiked Sorbent trap. The Sorbent traps were analyzed on site to determine the recovery of the spike and to determine if break through to the second section of each Sorbent trap had occurred. The recovery tests also verified that the sample rate and the sample time were sufficient to provide results within the calibration range of the analytical instrumentation.

During the Raw Mill On operating condition, four paired samples were collected simultaneously at the Scrubber Stack and at the Scrubber Inlet Duct at a sampling rate of approximately 0.500 liters per minute for sixty minutes. During the Raw Mill Off operating condition, five paired samples were collected simultaneously at the Scrubber Stack and at the Scrubber Inlet Duct at a sampling rate of approximately 0.600 liters per minute for forty-five minutes.

Samples were taken at a single traverse point (Port A, Point No. 3 at the stack and Port I Centroid Point at the inlet duct) during Run Nos. 1-4. Samples of forty-five minute duration were taken at a single traverse point (Port A, Point No. 3 at the stack and Port I Centroid Point at the inlet duct) during Run Nos. 5-9.

The Sorbent traps were leak checked at fifteen inches of mercury vacuum before each test, and again at the conclusion of each test. This was done to predetermine the possibility of a diluted sample.



The sampling train contained the following components:

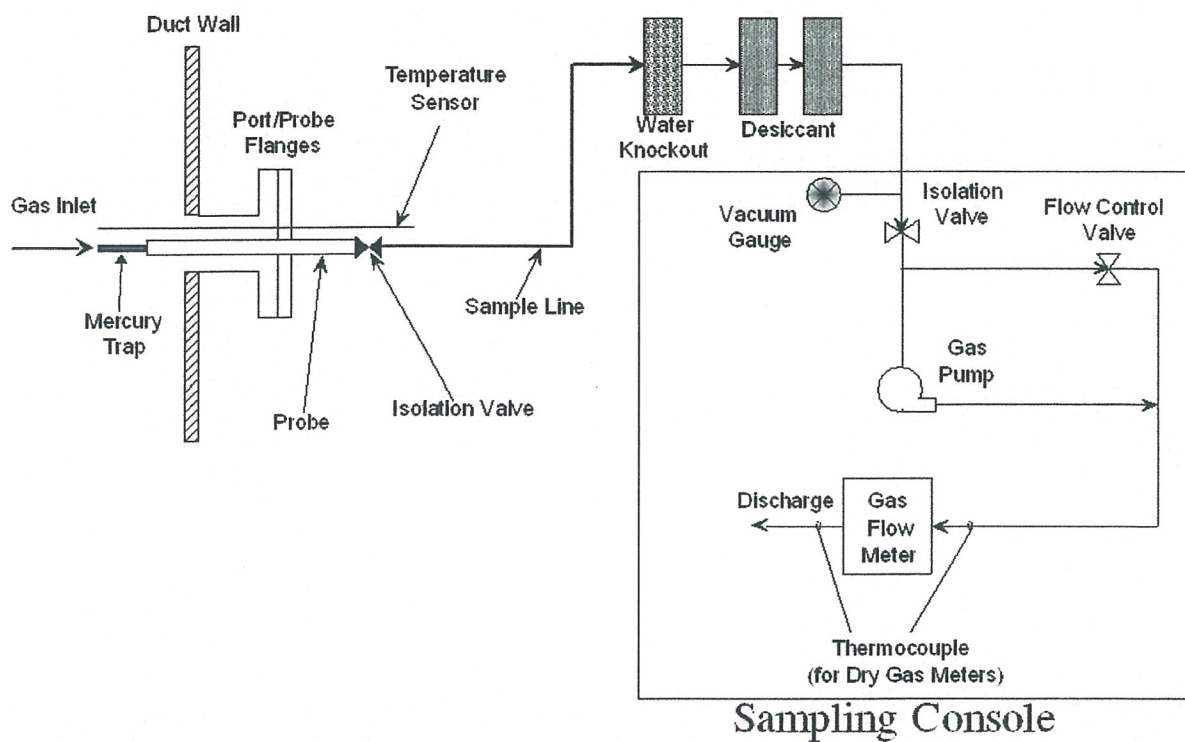
Sorbent trap heater @ 350°F

Heated stainless steel probe @ 350°F

Air cooled condenser system @ ambient temperature

At the conclusion of the tests, the samples were recovered and analyzed on site. The Ohio Lumex Company, Inc. analyzer was calibrated and each sample trap was analyzed and checked for break through to the second section of the trap. The Relative Deviation for each pair of Sorbent traps at each sampling location was also determined.

**Figure 5: EPA Draft Method 30B Sampling System**







**Flue Gas Adsorbent Mercury Speciation (FAMS)**

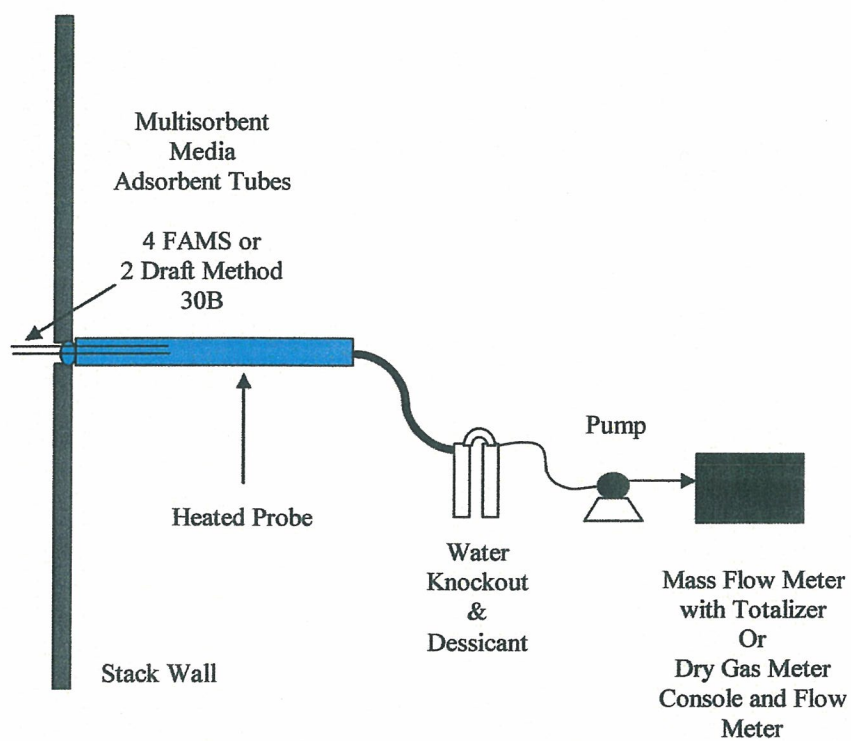
Speciated mercury sampling was performed according to the Frontier GeoSciences, Inc. Procedure, Flue Gas Adsorbent Mercury Speciation (FAMS). A copy of the procedure is included in Appendix H of this report.

The FAMS trains utilized two Sorbent traps per run at each location and the samples ran for sixty minutes during Run Nos. 1-4 and for forty-five minutes during Run Nos. 5-9 at a sampling rate of approximately 0.250 liters per minute. The Sorbent traps were leak checked before and after each run. The traps were located outside of the stack (within the probe) and maintained at a temperature of 177°C at the stack location and 95°C at the inlet location.

The samples on the Scrubber Stack were taken from a single point (Port D, Point No. 3). The samples on the Scrubber Inlet Duct were taken from a single point located at approximately the centroid of the duct, approximately 24 inches below the EPA Draft Method 30B sampling probe.

At the conclusion of each test run, the Sorbent traps were recovered and shipped to Frontier GeoSciences, Inc. for analysis.

**Figure 6: FAMS Sampling System**





## TEST NARRATIVE

Personnel from Air Sampling Associates, Inc. arrived at the Holcim (Texas) LP – Midlothian Plant, located in Midlothian, Texas at 6:45 a.m. on Tuesday, October 9, 2007. The sampling trailer was parked near the Desulphurization Scrubber Stack and the Desulphurization Scrubber Inlet Duct on Kiln No. 1 and power was supplied to the reference method analyzers. After a brief safety orientation, the sampling equipment was set-up on the Kiln No. 1 Scrubber Inlet Duct and the Scrubber Stack. Preliminary measurements were made and the equipment was secured. Personnel departed the plant at 2:45 p.m.

On Wednesday, October 10, 2007, personnel returned to the plant at 6:45 a.m. The reference method analyzers were calibrated and the sampling equipment was prepared for testing. The first simultaneous Recovery Test for EPA Draft Method 30B at the Scrubber Inlet Duct and the Scrubber Stack began at 8:52 a.m. Testing continued until the completion of the third simultaneous test at 12:45 p.m. The Sorbent tubes were recovered and analyzed. The sampling equipment was secured and personnel departed the plant at 2:00 p.m.

On Thursday, October 11, 2007, personnel returned to the plant at 6:45 a.m. The reference method analyzers were calibrated and the sampling equipment was prepared for testing. A stratification test was conducted at the Scrubber Stack. The first simultaneous test (Run No. 1) for mercury and oxygen at the Scrubber Inlet Duct and the Scrubber Stack during the Mill On test condition began at 10:30 a.m. Testing continued until the completion of the fourth test (Run No. 4) at 4:15 p.m. The first test (Run No. 1) for flow rate at the Scrubber Stack during the Mill On test condition began at 10:30 a.m. Testing continued until the completion of the fourth test (Run No. 4) at 4:15 p.m. Further testing was delayed when the Raw Mill ceased to operate. The





Sorbent tubes were recovered and analyzed. The sampling equipment was secured and personnel departed the plant at 6:45 p.m.

On Friday, October 12, 2007, personnel returned to the plant at 6:45 a.m. The reference method analyzers were calibrated and the sampling equipment was prepared for testing. The first simultaneous test (Run No. 5) for mercury and oxygen at the Scrubber Inlet Duct and the Scrubber Stack during the Mill Off test condition began at 8:00 a.m. Testing continued until the completion of the fifth simultaneous test (Run No. 9) at 1:45 p.m. The first test (Run No. 5) for flow rate at the Scrubber Stack during the Mill Off test condition began at 8:00 a.m. Testing continued until the completion of the fifth test (Run No. 9) at 1:45 p.m.

The Sorbent tubes were recovered and analyzed. The sampling equipment was moved off the Scrubber Inlet Duct and the Scrubber Stack and loaded into the sampling trailer. The FAMS tubes were shipped to Frontier GeoSciences, Inc. for analysis. The data was taken to Air Sampling Associates, Inc.'s office in Lewisville, Texas for further review.

Operations at the Holcim (Texas) LP – Midlothian Plant, Kiln No. 1 Desulphurization Scrubber Inlet Duct and Desulphurization Scrubber Stack located in Midlothian, Texas, were completed at 7:00 p.m., on Friday, October 12, 2007.